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Relationships between Organic Carbon and Total Organic Matter in Municipal Solid Wastes and City Refuse Composts

Emeterio Iglesias Jiménez & Víctor Pérez García

Instituto de Productos Naturales y Agrobiología de Canarias, CSIC, Avda. Francisco Sánchez 3, 38206 La Laguna, Tenerife, Canary Islands, Spain

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Abstract

The statistical study presented in this paper shows that no single factor can be shown to transform oxidizable carbon (OXC) into total organic carbon (TOC) when composts of different origin and decomposition degree are considered. Therefore, the Walkley–Black dichromatometric method is not suitable for the determination of TOC in municipal solid wastes (MSW) and city refuse composts (CRC). From the standard deviation of the sample population analysed, the degree of oxidizability of TOC may be deduced to fall within the range 66–98%.

Despite the great heterogeneity of the organic matter content of MSW and CRC, the TOC content remains constant around 54%, and a highly significant correlation ($p < 0{\cdot}001$) between total organic matter (TOM) and TOC has been deduced. The predictive equation ($TOM = 1{\cdot}135 + 1{\cdot}803TOC$, $r = 0{\cdot}968$, $n = 67$) may be very useful in commercial composting plants to deduce TOC (and assess the C/N ratio) from the simple determination of TOM by ignition loss. This procedure would avoid the use of a complicated apparatus to determine TOC and a longer determination time. In general, a factor of 1·8 may be assumed for practical application.

Key words: Municipal solid waste, city refuse compost, biowastes, carbon, total organic matter, oxidizability degree, conversion factor, composting control parameters.

INTRODUCTION

The C/N ratio is one of the most important parameters in control of the composting process and in determination of the degree of maturity of the newly formed organic materials (Iglesias Jiménez & Pérez García, 1989). The suitable C/N ratio of the initial material should be 26–35 to ensure a good composting rate (Mote & Griffis, 1980). A C/N ratio below 20 is indicative of an acceptable maturity in the final product, a ratio of 15 or even less being preferable (Inbar *et al.*, 1990*a*). Traditionally, the determination of total organic carbon (TOC) for the assessment of the C/N ratio is performed following two procedures: dichromatometric oxidation (Walkley & Black, 1934) and direct determination by dry combustion in a C-analyser (N normally by the Kjeldahl procedure). The former gives only the oxidizable carbon and it is necessary to apply a suitable recovery factor. The conventionally accepted 77% recovery for soils is not readily applicable due to the different degrees of oxidation of the organic matter of compost decomposed to varying degrees (Hernández *et al.*, 1988). The latter procedure requires the use of a complicated apparatus and a greater time of determination.

TOC may also be deduced indirectly from the total organic matter TOM) content determined by ignition loss, applying a suitable factor of transformation. In soil organic matter studies the 'Van Bemmelen' factor of 1·724 is commonly used, based on the assumption that humified organic matter of soil contains approximately 58% C

(Nelson & Sommers, 1982). However, a considerable variation exists in the TOM/TOC ratio among different soils, and also among horizons of the same soil (Allison, 1965). Thus, it is presently considered that its application is not justified for every kind of soil (Nelson & Sommers, 1982) and it is only used as a conventional factor.

The nature of the organic matter of composts, manures and other newly formed organic materials is different from that of soil organic matter, particularly the humic-like fractions (González-Vila & Martín, 1985; Hernández *et al.*, 1990; Inbar *et al.*, 1990*b*). Therefore, the conversion factor for soils is even less applicable for these materials (Navarro *et al.*, 1990).

The aim of the present work was, therefore, to deduce a predictive equation and a tentative conversion factor for the assessment of TOC (and C/N ratio) from the TOM determination by ignition loss for a practical application in commercial composting plants.

METHODS

Samples

The samples used in the present study are listed in Table 1 and have the following origins.

(a) Samples 1–28: organic fraction of municipal solid wastes (MSW_0)

These samples correspond to a study performed on the physical characterization and chemical composition of MSW from Tenerife Island. MSW was collected at 28 different points in the capital city, Santa Cruz de Tenerife. Since one of the most noteworthy characteristics of MSW is their great heterogeneity, each sample was composed of a large amount of refuse material, approximately 600 kg. Contaminating materials such as glass, gravel, plastics, cardboard and metal were separated by hand for classification purposes, leaving paper only. The organic fraction was air-dried and submitted to a second removal of small fragments of inert materials. The residue was ground in a hammer mill (mesh size 2 cm) and thoroughly mixed. This material was spread to form a circle approximately 2 m in diameter and divided into quarters. Two opposite sectors were taken to form a new circle after mixing. Four final samples for analysis (1 kg) were obtained after sequential fractionation following this procedure. The analytical data given in Table 1 are the average of these four repetitions.

(b) Composting piles (CP)

Samples 29–48 were obtained periodically during two controlled piling trials with 75 days' duration of the biooxidative phase and 90 days of complementary maturation: an R-pile, exclusively formed by Tenerife MSW_0 (810 kg), and an R+S-pile, formed by a mixture of MSW_0 (850 kg) and dehydrated sewage sludge (370 kg). Each sample (3 kg) was composed of eight combined subsamples taken from the surface of the pile just formed after the turning-over operation. A total of three repetitions was taken each time (24 subsamples). The analytical data given in Table 1 are the average of these three repetitions. Detailed descriptions of the composting process have been reported in previous papers (Iglesias Jiménez *et al.*, 1986; Iglesias Jiménez & Pérez García, 1991, 1992).

(c) City refuse composts (CRC)

Samples 49–67 are final products obtained from several different factories in Spain (samples 49–63), Switzerland, Belgium and Germany.

Analytical determinations

All the samples were dried at 65°C for 48 h in a forced-draught cabinet. In CP and CRC samples the inert material was removed. Special care was then taken to eliminate totally the fragments of plastics remaining from all the samples, because the inert-C of plastics biases the oxidizable carbon (OXC) and TOC results (Juste, 1980). This material was crushed in a hammer mill (mesh size 2 mm) followed by further pulverization using a vibrating cutter, and analysed.

Total organic matter (TOM) was determined by ignition loss at 600°C, as recommended by Gallardo *et al.* (1987). The determination of TOM by calcination is not really accurate since there exist volatile mineral compounds that prevent the value obtained by calcination from being considered as true organic matter (Brame & Lefevre, 1977; Moré *et al.*, 1987). This procedure therefore provides an approximation, slightly in excess of the true level of TOM. However, from the point of view of practical application for commercial composting plants this difference may be considered to be negligible, due to the high organic content of these materials. This is the criterion followed in this paper.

Total carbon was determined by combustion in a Wösthoff (Carmhograph *12-H Omega*) carbon analyser. Total organic carbon (TOC) was

Table 1. Mean values of total organic matter (TOM), total organic carbon (TOC), oxidizable carbon (OXC) and transformation factors (TOC to TOM, OXC to TOC) of all the samples analysed

(a) Tenerife MSW_o

Sample	*TOM*	*TOC*	*OXC*	*TOM/TOC*	*TOC/OXC*
1	85·2	46·8	36·5	1·82	1·28
2	87·4	48·1	38·4	1·82	1·25
3	89·7	45·8	36·9	1·96	1·24
4	83·2	48·2	35·9	1·73	1·34
5	82·8	45·0	19·7	1·84	2·29
6	79·2	38·8	19·0	2·04	2·04
7	76·6	43·6	33·8	1·76	1·29
8	72·3	40·0	26·8	1·81	1·49
9	82·3	47·6	45·7	1·73	1·04
10	89·9	47·1	35·3	1·91	1·33
11	87·1	47·5	30·7	1·83	1·55
12	92·0	48·3	30·6	1·91	1·58
13	86·0	45·7	35·7	1·88	1·28
14	83·1	40·8	32·5	2·04	1·25
15	92·3	44·8	34·8	2·06	1·29
16	78·8	45·5	39·2	1·73	1·16
17	88·4	48·0	36·9	1·84	1·30
18	78·2	40·8	34·2	1·92	1·19
19	83·1	47·8	38·0	1·74	1·26
20	76·1	41·4	34·5	1·84	1·20
21	70·1	40·8	36·3	1·72	1·12
22	85·3	43·1	35·2	1·98	1·22
23	70·0	36·0	26·0	1·94	1·38
24	72·2	41·5	33·0	1·74	1·26
25	68·5	37·0	30·3	1·85	1·22
26	70·0	39·8	35·3	1·76	1·13
27	65·4	34·1	30·3	1·92	1·13
28	87·1	44·7	41·0	1·95	1·09

(b) CP (R-pile)

Sample	*Days*	*TOM*	*TOC*	*OXC*	*TOM/TOC*	*TOC/OXC*
29	1	86·2	48·3	45·2	1·78	1·07
30	9	85·5	45·5	43·4	1·88	1·05
31	16	80·6	45·3	42·2	1·78	1·07
32	23	77·3	44·5	42·5	1·74	1·05
33	35	71·8	41·3	40·3	1·74	1·02
34	43	69·6	40·0	37·1	1·74	1·08
35	51	69·6	37·2	36·3	1·87	1·02
36	60	68·5	36·8	33·0	1·86	1·12
37	68	69·0	36·6	32·7	1·89	1·12
38	75	68·8	35·6	31·9	1·93	1·12
39	165	61·3	32·6	31·0	1·88	1·05

CP (R + S-pile)

40	1	74·6	42·8	38·6	1·74	1·11
41	10	66·2	37·8	35·8	1·75	1·06
42	18	62·9	34·7	33·4	1·81	1·04
43	25	62·7	33·3	33·0	1·88	1·01
44	34	58·6	32·3	29·2	1·81	1·11
45	46	57·5	32·0	28·3	1·80	1·13
46	62	51·9	29·4	21·8	1·77	1·35
47	75	50·1	28·9	20·9	1·73	1·38
48	165	43·6	25·1	21·7	1·74	1·16

(c) CRC

Sample	*Origin*	*TOM*	*TOC*	*OXC*	*TOM/TOC*	*TOC/OXC*
49	Barcelona	58·0	33·6	29·2	1·73	1·15
50	Barcelona	59·6	28·1	23·4	2·12	1·20
51	Mataró	45·8	24·0	22·2	1·91	1·08
52	Mataró	49·9	22·7	18·7	2·20	1·21

Table 1. — *contd.*

Sample	*Origin*	*TOM*	*TOC*	*OXC*	*TOM/TOC*	*TOC/OXC*
53	Villafranca	68·3	37·5	20·7	1·82	1·81
54	Villafranca	54·4	33·3	28·6	1·63	1·16
55	Tarragona	34·7	18·7	14·5	1·86	1·29
56	Seville	34·0	18·1	16·8	1·88	1·08
57	Seville	44·3	20·0	14·3	2·21	1·40
58	Seville	39·7	22·9	14·8	1·73	1·55
59	Granada	56·3	31·8	15·9	1·77	2·00
60	Las Palmas	51·3	28·2	22·5	1·82	1·25
61	Murcia	50·1	32·3	28·7	1·55	1·13
62	Madrid	49·7	30·7	27·3	1·62	1·12
63	Madrid	39·4	23·5	16·8	1·68	1·40
64	Switzerland	53·2	29·7	27·8	1·79	1·07
65	Switzerland	52·3	27·8	17·2	1·88	1·62
66	Germany	47·4	26·3	22·2	1·80	1·18
67	Belgium	31·6	16·4	12·3	1·93	1·33

MSW_0, Organic fraction of the municipal solid wastes of Tenerife.
CP, Samples obtained periodically from a composting–piling trial with city refuse (R-pile) and city refuse + sewage sludge (R + S-pile).
CRC, City refuse composts (final products) from several European factories.

deduced by subtracting the inorganic carbon (calcimeter test) from total carbon.

Oxidizable carbon (OXC) was determined by the modified dichromatometric oxidation method of Walkley and Black (1934) (W–B method, $K_2Cr_2O_7 + H_2SO_4$ mixture), based on the external use of heat up to a temperature of 150°C for one minute (US Salinity Laboratory Staff, 1954). In soil samples it has been found that this modified wet-digestion method gives approximately 89% recovery of TOC, as compared to the dry-combustion method (Allison, 1965).

Results of chemical analysis of MSW_0 and compost materials are expressed on a dry weight basis (105°C, 4 h). The analysis of the replicate samples of MSW_0 and PC was carried out in duplicate and that of the CRC samples in triplicate.

Statistical analysis

All the statistical procedures of this paper (population sample distribution, analysis of variance, regression analysis, covariance analysis) have been performed according to the methods described by Snedecor and Cohran (1967). The Statistical Graphics System Program (STSC) was used to calculate F values, Tukey's multiple range test, regression equations and correlation coefficients relating OXC, TOC, TOM and transformation factors.

RESULTS AND DISCUSSION

In Table 1 the mean values of TOM, OXC and TOC of all the samples analysed are shown. The ratios TOC/OXC and TOM/TOC are also indicated.

Relationship between OXC and TOC (recovery factor)

The mean conversion factors of OXC to TOC (f average) and a statistical summary of the three groups of samples are shown in Table 2. The MSW_0 and CRC groups show a very high coefficient of variation (CV), around 20%. The CV of the CP group may be considered as acceptable (< 10%) and may be explained because the samples were produced from identical raw materials. One-way analysis of variance (ANOVA) gives an F ratio of 6·47 which is significant (d.f.: 2·64; significance level: 99·72%) and the Tukey range test shows that the f average of PC is significantly lower than MSW_0 and CRC f averages. Thus, the average factor is different for different groups of materials.

Considering total samples ($n = 67$), a great dispersion of the data with respect to the mean is observed (standard deviation: 0·247; coefficient of variation: 19·6%). From the standard deviation a range of 66–98% of TOC recovery may be deduced, although values between 44 and 99% have been observed. Therefore, the global average is not statistically acceptable. This fact may be explained by the characteristically high heterogeneity of these materials, which contain a wide range of carbonaceous compounds whose relative amounts can vary considerably according to their geographic origin. Thus, Moré *et al.* (1987) reported that only a mean value of 30% of

the total-C (Carmhograph) was the amount oxidized with dichromate in 20 samples of compost. In these materials, the content in plastic fragments can considerably influence the oxidation per cent. As shown by Juste (1980), only 1–15% of the C content in plastics is destroyed by chemical oxidants such as potassium dichromate. The samples must therefore be thoroughly freed of plastics prior to their analysis, as performed in this work.

Table 3 gives the equations deduced for each group and a statistical summary for each correlation. The equation of MSW_0 is significant at the 95% level (F ratio: 8·1; d.f.: 1, 26). The equations of CP and CRC show a high coefficient of correlation, significant at the 99·9 and 99% levels, respectively. The test for the homogeneity of the slopes gives an F value of 11·05, which is significant (d.f.: 2, 61) and indicates that the three slopes are different. Thus, despite the fact that the correlation between TOC and OXC is significant, the regression equation for each group of samples is statistically different, and a general equation is not possible.

On the basis of the samples analysed, it can be seen that the W–B method is not suitable for TOC determination. The conventionally accepted factor for soils $f = 1{\cdot}3$ (77% of TOC recovery) for the method based on the heat of dilution and $f = 1{\cdot}12$ (89%) with external heat (150°C, 1 min) are not applicable to organic materials of an urban nature. The results may be presented as uncorrected values (oxidizable carbon). Thus the C/N ratio (TOC/N–Kjeldahl) cannot be established by this procedure.

It should be noted that this procedure for establishing TOC of city refuse composts and compost-amended soils is reported in a great number of articles. As shown by Hernández *et al.* (1988), the single 77% recovery factor (W–B method) does not seem appropriate either for soils recently treated with a range of organic materials, already decomposed to varying degrees. Moré *et al.* (1987) showed that the oxidation degree depends on the chemical nature of the organic compounds, especially the quantity of aromatic molecules, nitrogen-heterocyclic groups and polymerization degree. Indeed, Bornemisza *et al.* (1979) indicated that the W–B method should not be employed for organic C studies in deeper layers of natural soils. These authors proved that organic matter in the deeper horizons is more easily oxidized, perhaps because fulvic acids with low molecular weights are more abundant. In some cases, oxidation is almost complete. Richter *et al.* (1990) also reported that the degree of oxidation with dichromate may vary in natural soils from 75 to 90%. Finally, Pratt and Sánchez (1973) demonstrated that the degree of oxidation depends on the total carbon content of soil, particularly in acid soils.

Relationship between TOC and TOM (transformation factor)

In Table 4 the average value of the TOM/TOC ratio (f average) and statistics are shown. The CV

Table 2. Recovery factors (TOC/OXC ratio) and statistical summary of the organic fraction of municipal solid wastes (MSW_0); samples taken from a composting–piling trial (CP) and city refuse composts from several European factories (CRC)

Samples	*n*	*TOC/OXC ratio*	*Range*	*SD*	*V*	*CV (%)*	*TOC recovery (%)*
MSW_0	28	1·329	1·04–2·29	±0·269	0·072	20·2	75·2
CP	20	1·105	1·01–1·38	±0·098	0·010	9·3	90·5
CRC	19	1·318	1·07–2·00	±0·259	0·067	19·7	75·9
Total samples	67	1·259	1·01–2·29	±0·247	0·061	19·6	79·4

SD, Standard deviation; V, variance; CV, coefficient of variation.

Table 3. Correlations for total organic carbon (TOC) versus oxidizable carbon (OXC)

Samples	*n*	*Equation*	*r*	*F ratio*	*p*	*SE of estimate*
MSW_0	28	$Y = 31{\cdot}863 + 0{\cdot}346X$	0·488	8·12	<0·05	3·61
CP	20	$Y = 8{\cdot}481 + 0{\cdot}841X$	0·966	255·21	<0·001	1·61
CRC	19	$Y = 10{\cdot}668 + 0{\cdot}769X$	0·725	18·80	<0·01	4·20

MSW_0, Organic fraction of municipal solid wastes; CP, composting piles; CRC, city refuse composts.

Table 4. Factors of transformation (TOM/TOC ratio) and statistical summary of the organic fraction of municipal solid wastes (MSW_0); samples taken from a composting–piling trial (CP), and city refuse composts from several European factories (CRC)

Samples	*n*	*TOM/TOC ratio*	*Range*	*SD*	*V*	*CV (%)*	*C-content (%)*
MSW_0	28	1·859	1·72–2·06	±0·103	0·011	5·54	53·8
CP	20	1·806	1·73–1·93	±0·065	0·042	3·61	55·4
CRC	19	1·838	1·55–2·21	±0·182	0·033	9·91	54·4
Total samples	67	1·837	1·55–2·21	±0·123	0·015	6·70	54·4

SD, Standard deviation; *V*, variance; CV, coefficient variation.

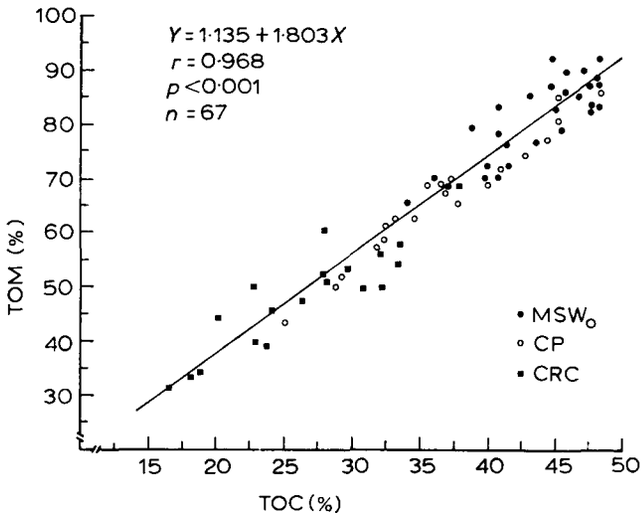

Fig. 1. Relationship between total organic matter (TOM) and total organic carbon (TOC). MSW_0, Organic fraction of the municipal solid wastes of Tenerife Island; CP, samples obtained periodically from two composting–piling trials; CRC, city refuse composts from several European countries.

of the three groups is acceptable (< 10%). The low dispersion of the value of *f* for MSW_0 is particularly remarkable. Normally, the composition of MSW varies greatly, depending on the sorting policies of the city refuse system and, above all, on the different wealth of the citizens and their eating habits. Despite this variability, the low CV of MSW_0 (5·5%) suggests that a constancy exists in the TOC% of the organic matter when considering a single city.

The analysis of variance *F* value was not significant (*F*: 1·08; d.f.: 2, 64; significance level: 65·3%) and thus the three mean values are statistically equal.

Considering total samples ($n = 67$), an average value of 1·837 ± 0·123 is deduced. The CV is very low (6·7%) and the population shows a good normal distribution (geometric mean: 1·833; median: 1·821; mode: 1·821). Thus, this factor may be considered statistically acceptable for the set of samples analysed. From the standard deviation it may be deduced that the organic matter of these materials, of a different origin and degree of decomposition, normally has a carbon content between 51 and 58% (average: 54%). The mean value is only slightly lower than that conventionally accepted for soils, 58%.

The correlations for MSW_0, CP and CRC are similar in form (Table 5). The test for the homogeneity of the slopes gives an *F* value of 2·27, which is not significant (d.f.: 2, 61). Therefore, the three slopes are statistically equal, and a general equation may be assumed. The figure shows the linear regression and equation using all the data points. It is significant at the 99·9% level ($r = 0·968$, $n = 67$).

This equation allows TOC to be calculated from the simple determination of TOM by ignition loss. This procedure, due to its simplicity, may be used in commercial composting plants instead of the direct determination of C. Moreover, the determination of TOM by calculation allows a large amount of material to be analysed and therefore minimizes error due to the heterogeneity of the material.

CONCLUSIONS

The results presented in this paper suggest that the variability of the oxidation of the organic matter from municipal solid wastes and city refuse composts is a reflection of their great heterogeneity. Therefore, a single predictive equation cannot be deduced to transform oxidizable carbon into total organic carbon, when materials of a different degree of decomposition are considered. The assessment of TOC (and the C/N ratio) by means of the W–B dichromatometric method (oxidizable carbon × recovery factor) is not suitable for urban composts. The results

Table 5. Correlations for total organic matter (TOM) versus total organic carbon (TOC)

Samples	*n*	*Equation*	*r*	*F ratio*	*p*	*SE of estimate*
MSW_o	28	$Y = 11{\cdot}599 + 1{\cdot}590X$	0·832	58·47	<0·01	4·39
CP	20	$Y = 1{\cdot}736 + 1{\cdot}759X$	0·970	381·90	<0·001	2·45
CRC	19	$Y = 10{\cdot}059 + 1{\cdot}442X$	0·899	72·14	<0·01	4·27
Total samples	67	$Y = 1{\cdot}135 + 1{\cdot}803X$	0·968	977·14	<0·001	4·10

MSW_o, Organic fraction of municipal solid wastes; CP, composting piles; CRC, city refuse composts.

should be reported as uncorrected values of oxidizable carbon only.

However, despite the heterogeneity of the organic composition of these materials, the statistical study performed in this paper shows that a constancy exists in the TOC content of TOM (around 54%), and the factor of transformation does not depend on the degree of maturity. The predictive equation deduced (TOM = 1·135 + 1·803 TOC or TOC = 1·703 + 0·520 TOM) may prove very useful in commercial composting plants. In general, a factor of 1·8 may be assumed for a practical application.

The results described in this paper are necessarily provisional and more overall data, from other parts of the world, would be necessary to confirm them. Moreover, the predictive equation can be considered valid only for city refuse composts of which the organic fraction of municipal solid wastes is the raw material for composting.

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